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Crack Growth Testing of an Aluminum Oxynitride (AlON) for International Space Station Kick Panes

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Summary

The mechanical properties of an aluminum oxynitride supplied as ground beams and disks were measured using ASTM International (formerly American Society for Testing and Materials) standard test methods. The slow crack growth tests were complicated by a “short” finish that increased strength scatter. Refining of the finish by more material removal in the second stage of grinding or the use of uniaxial grinding as specified in ASTM C1499 might have avoided the issue. The structural design parameters are an elastic modulus of $E = 319$ GPa, Poisson’s ratio of $\nu = 0.26$, a fracture toughness of $K_{Icb}(A) = 2.18$ MPa \sqrt{m} , slow crack growth (SCG) parameter $n = 36$, and SCG parameter $A = 1.96 \times 10^{-11}$ m/s(MPa \sqrt{m}) n . For a ground finish, the Weibull parameters are a mean modulus of $m = 14.0$ and characteristic strength of $\sigma_0 = 250.2$ MPa. The 2015 vintage material exhibits similar mechanical properties to a 2010 vintage billet. Indentation flaws were not sensitive to the inherent crack growth mechanisms of this material and produced misleading results.

Introduction

Aluminum oxynitride (ALON) spinel is a relatively new, transparent polycrystalline ceramic, which can be processed into relatively large, dense components. Its hardness and high fracture toughness relative to other transparent materials makes ALON applicable to impact-resistant, specialty windows (Ref. 1).

The interior panes of the International Space Station (ISS) windows are protected by borosilicate glass “kick” panes. Over time, the optical condition of the kick panes has degraded and replacement is being considered. Although the current material could be used for replacement panes, tougher materials have been developed. This report summarizes the mechanical testing of ALON[®] (Surmet Corporation), a relatively new, commercial ALON being considered for kick panes. This testing is being done in conjunction with optical and impact testing being performed at NASA Johnson Space Center, Kennedy Space Center, and Langley Research Center.

Mechanical Properties

Elastic Modulus

The elastic modulus and Poisson’s ratio were measured using impulse excitation in accordance with ASTM C1259 (Ref. 2). The measured values are $E = 319 \pm 1$ GPa and $\nu = 0.263 \pm 0.001$. This is comparable to measurements on a 2010 vintage billet of ALON[®] (314 ± 1 GPa, $\nu = 0.26$) (Ref. 3).

Fracture Toughness

Fracture toughness was measured by using the chevron-notched beam (VB) in accordance with ASTM C1421 (Ref. 4). The results are summarized in Tables I and II. The measured value of $K_{Icb}(A) = 2.19 \pm 0.04$ MPa \sqrt{m} in dry N₂ is in agreement with that measured on a 2010 vintage billet of ALON[®] (2.18 ± 0.14 MPa \sqrt{m}) (Ref. 3). Several SEPB (single-edge-precracked-beam) tests were also run in lab air

on the 2010 material, resulting in $K_{Ipb} = 2.02 \pm 0.04 \text{ MPa}\sqrt{\text{m}}$. Figure 1 shows a SEPB fracture surface that indicates that transgranular fracture occurs during rapid crack propagation.

In comparison, the fracture toughness of borosilicate glass is about one-third that of ALON[®], at $0.72 \pm 0.04 \text{ MPa}\sqrt{\text{m}}$ (Ref. 5).

Strength and Slow Crack Growth

Slow crack growth (SCG) properties were measured by using ASTM C1368 (Ref. 6). The disk test specimens appear to have been ground in two stages. The second stage removed the first, likely Blanchard, grinding stage to varying degrees, as shown in Figure 2. This resulted in a “short” finish and two distinct flaw populations. As a result, specimens occasionally failed from first stage scars, thereby leading to scatter in measured strength and a coefficient of variation (CV) of ~14 percent at each stress rate. A secondary factor that may have led to strength scatter is the coarse grain size. To get a better estimate of the SCG parameters, specimens that failed from the Blanchard-type scratches shown in Figure 2 were censored for the SCG data analysis. This reduced the CV at each rate to ~10 percent, comparable to that of the 2010 set, which exhibited a CV of ~9 percent. Resultant, censored, and unbiased Weibull parameters were a median modulus of $m = 14.0$ and characteristic strength of $\sigma_0 = 250.2 \text{ MPa}$. As the data needed to be censored, the RBA (reduced bias adjustment of Abernethy) (Ref. 7) rather than the TBA (Thoman, Bain, Antle) method (Ref. 8) of ASTM C1239 (Ref. 9) was employed. However, the TBA method resulted in a similar mean modulus of $m = 13.6$.

The resultant stress rate curve is shown in Figure 3, and the crack velocity curves are shown in Figure 4 for the power and exponential functions

$$v = \frac{da}{dt} = A_1 K_I^{n_1} = A_1^* \left[\frac{K_I}{K_{Ic}} \right]^{n_1} \quad (1)$$

and

$$v = \frac{da}{dt} = A_2 \exp(n_2 K_I) = A_2 \exp\left(n_2^* \frac{K_I}{K_{Ic}}\right) \quad (2)$$

where v , a , and t are crack velocity, crack size, and time, respectively. Constants A and n are the material/environment dependent SCG parameters, and K_I and K_{Ic} are, respectively, the Mode I stress intensity factor and the critical stress intensity factor or fracture toughness. The parameters are summarized in Tables III to IV for units common to the engineering literature and those used in NASGRO[®] (Southwest Research Institute (SWRI)). These results are similar to those from a 2010 billet ($n = 33 \pm 5$) (Ref. 3). In comparison to borosilicate glass ($n \approx 17$; $K_{Ic} \approx 0.72 \text{ MPa}\sqrt{\text{m}}$), the higher fracture toughness and n value of ALON[®] results in much lower crack velocities at a stress intensity, as shown in Figure 4. For typical glasses, $n \approx 12 - 20$ and $K_{Ic} \approx 0.6 - 0.8 \text{ MPa}\sqrt{\text{m}}$ (Ref. 5), substantially less than that of ALON[®].

In addition to testing ground specimens, a series of tests were conducted by using specimens damaged via Vickers indentation. This consistently produced failure at the indentation, and because of the large grain size, this failure was typically within a single grain or two adjoining grains, as shown in Figure 5. This approach produced relatively little strength loss as a function of stress rate as shown in Figure 6, with $n > 86$ as shown in Figure 4.

The lack of strength loss is likely a result of poor sampling of the material microstructure by use of a single crack. The use of grinding samples the microstructure better by producing microcracks across the whole specimen surface, thereby allowing damage to evolve naturally. Indentation cracks, though large,

are much smaller than the grains and do not effectively sample the behavior of inherent flaws or that of a multiplicity of grinding flaws, but instead the properties of a grain in random, rather than worst, orientation. An example of an inherent flaw (a region of porosity), which indentation flaws have difficult sampling, is shown in Figure 7. This result is in contrast to the successful use of indentations in the testing of glasses (Ref. 5).

Fracture Branch Constants

The fracture branching radii were measured on test specimens stressed at several rates. Because the strengths were narrowly distributed, the mirror constants were estimated pointwise from, rather than by curve fitting of, the function

$$S_f = \frac{A_i}{\sqrt{r_i}} \quad (3)$$

where A_B is the branch constant and r_B is the corresponding branch radius. The resultant constants are tabulated in Table VI. The estimated A_B values show a dependence on rate and environment. This can be interpreted as a dependence of A_B on branch size, stored energy level, or load rate, as all are correlated or inversely correlated. For the combined data sets, $A_B = 8.1 \pm 1.3 \text{ MPa}\sqrt{\text{m}}$.

Conclusions

The elastic constants, fracture toughness, and slow crack growth parameters of ALON[®] (Surmet Corporation) were measured by using ASTM International (formerly American Society for Testing and Materials) methods. The structural design parameters are an elastic modulus of $E = 319 \text{ GPa}$, Poisson's ratio of $\nu = 0.26$, a fracture toughness of $K_{Ivb} = 2.18 \text{ MPa}\sqrt{\text{m}}$, slow crack growth (SCG) parameter $n = 36$, and SCG parameter $A = 1.96 \times 10^{-11} \text{ m/s}(\text{MPa}\sqrt{\text{m}})^{-n}$. For a ground finish, the Weibull parameters were a mean modulus of $m = 14.0$ and characteristic strength of $\sigma_0 = 250.2 \text{ MPa}$. The material exhibits similar mechanical properties to a 2010 vintage billet. Indentation flaws were not sensitive to the inherent crack growth mechanisms of this material and implied little slow crack growth. Caution should be taken when using indentation flaws to characterize materials with a coarse grain structure because the inherent crack growth behavior may not be characterized. As compared to borosilicate glass, ALON[®] has three times the fracture toughness and twice the slow crack exponent.

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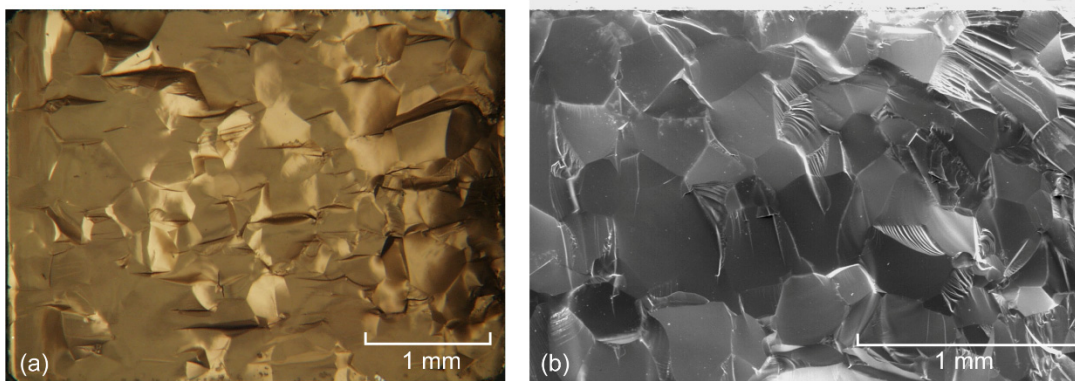


Figure 1.—Fracture surfaces. (a) Optical view of a single-edge-precracked-beam (SEPB).
(b) Scanning electron view of a slow crack growth specimen.

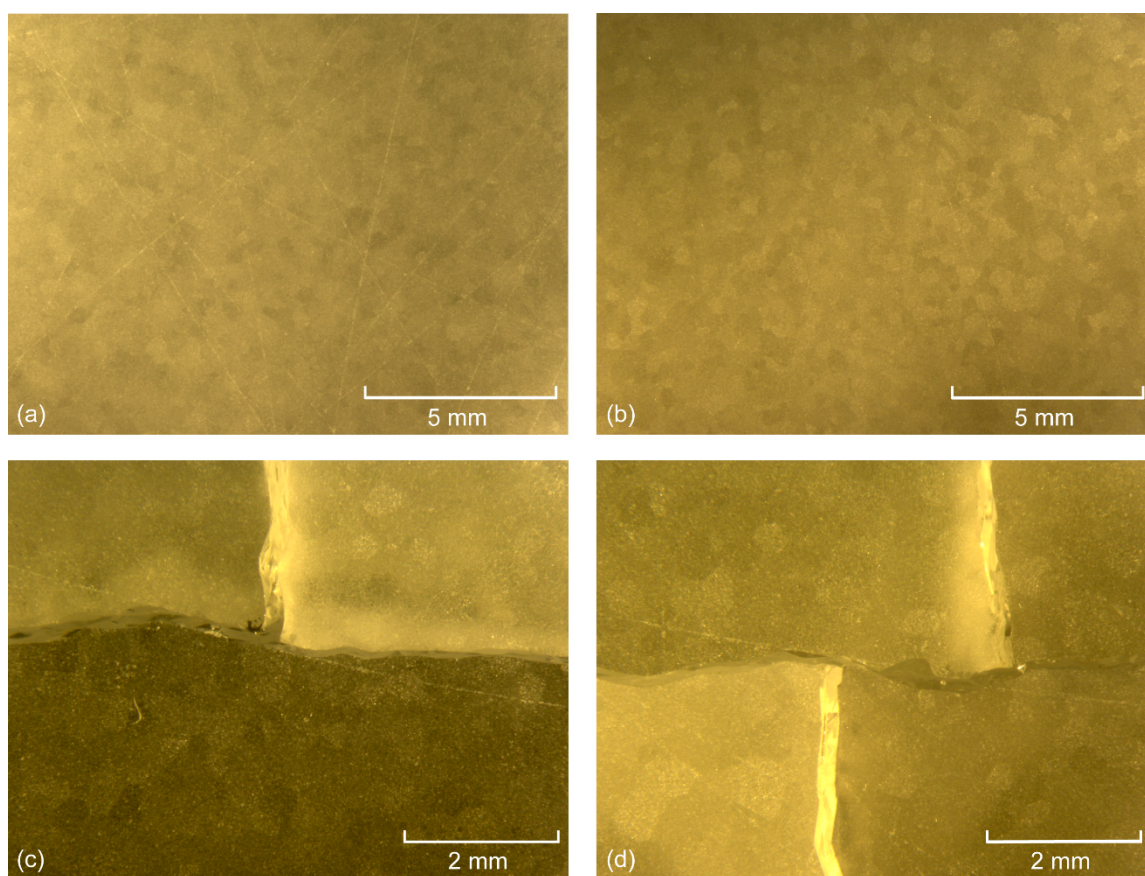


Figure 2.—Grinding marks in two different test specimens. (a) Frequent scratches ($\times 7$). (b) Shallow and infrequent scratches ($\times 7$). (c) Failure at an obvious scratch at 0.23 MPa/s ($\times 15$). (d) Failure at an obvious scratch at 0.0023 MPa/s ($\times 15$).

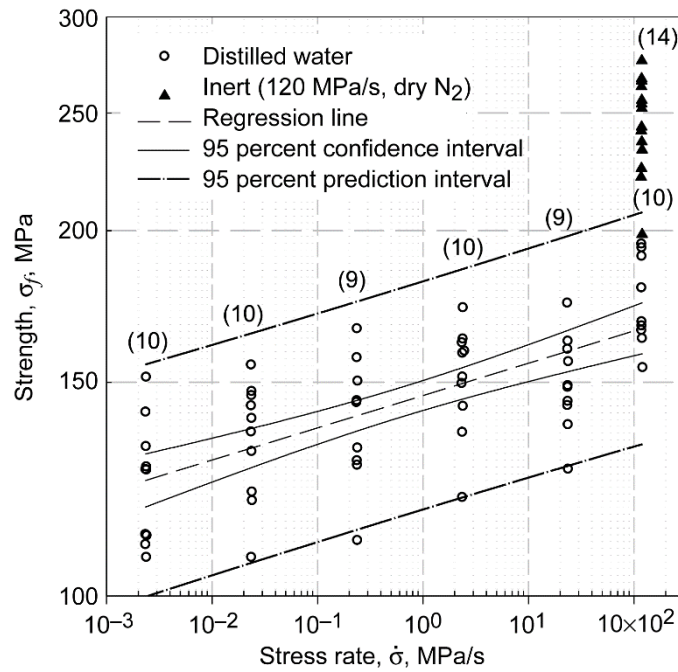


Figure 3.—Fracture strength as a function of stress rate for ground specimens. The number of specimens tested is given in parentheses.

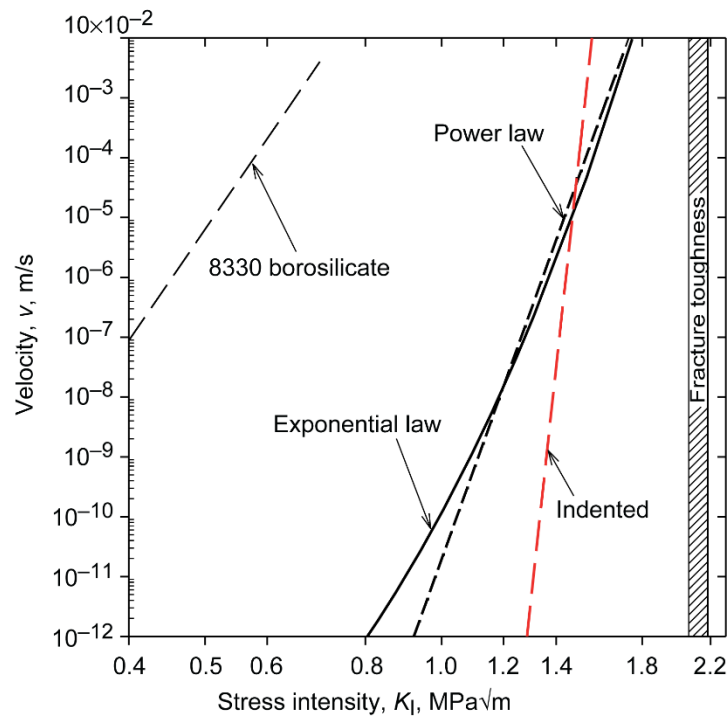


Figure 4.—Crack velocity of ALON® in water and 8330 borosilicate glass in 95 percent relative humidity as a function of stress intensity.

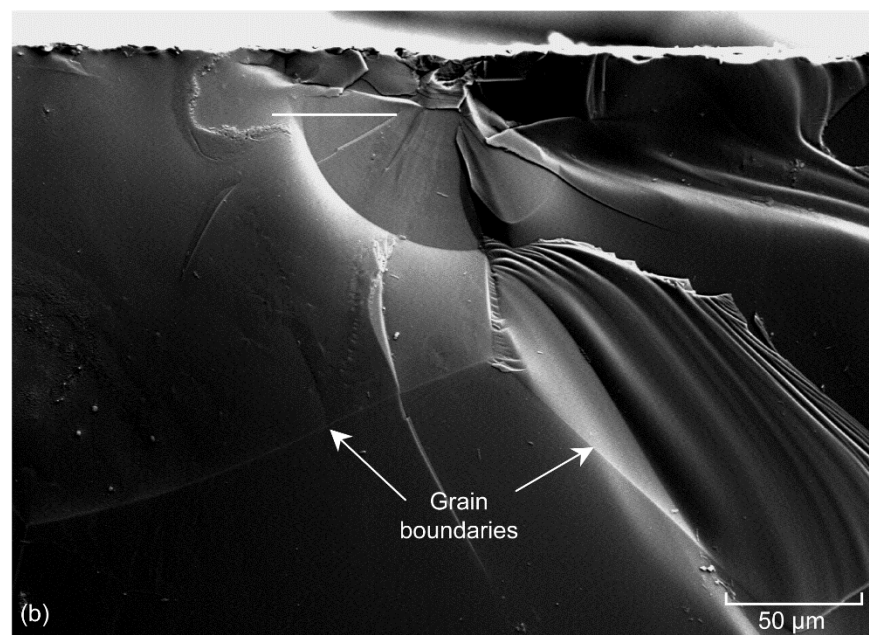
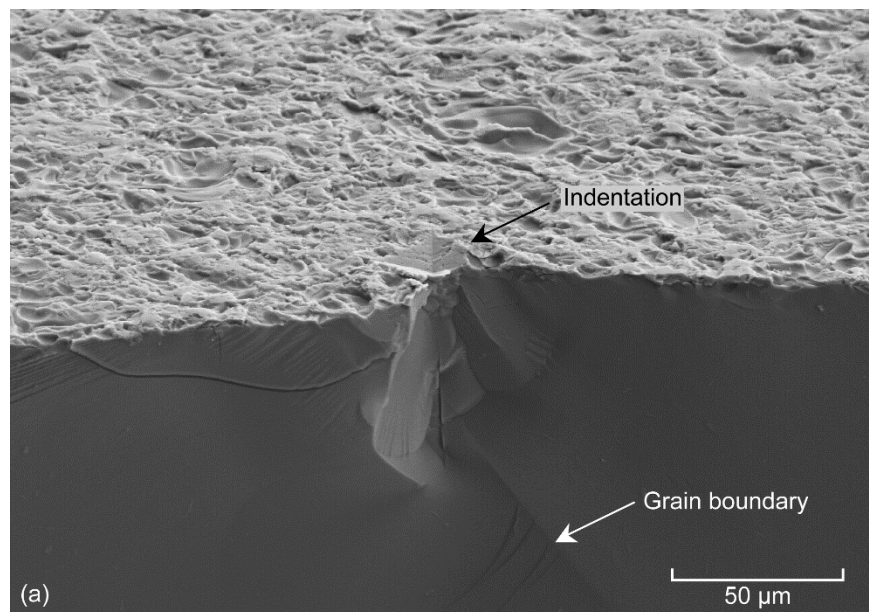


Figure 5.—Indentation cracks.

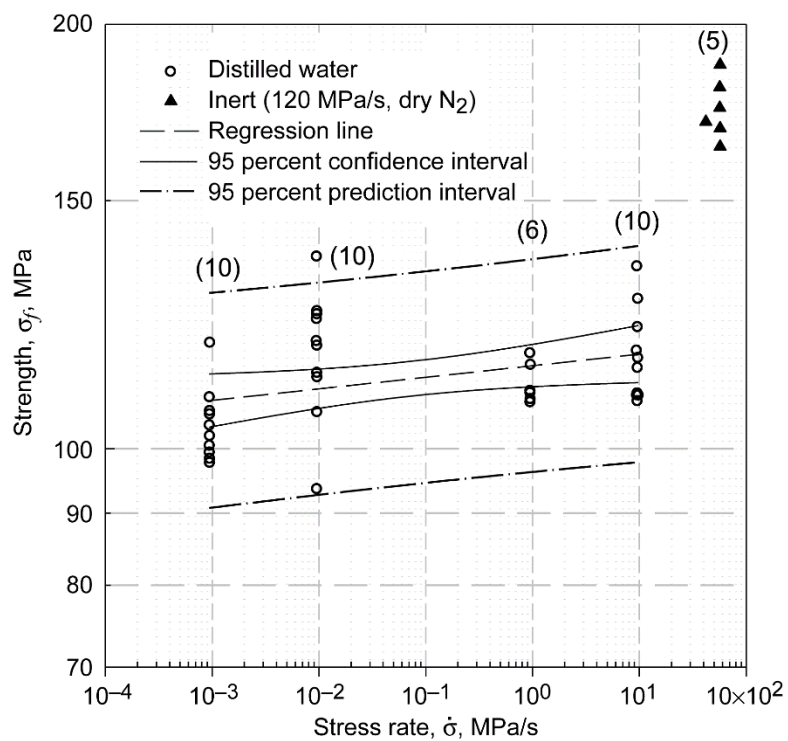


Figure 6.—Strength as a function of stress rate for indented ALON[®] specimens. The number of specimens tested is given in parentheses.

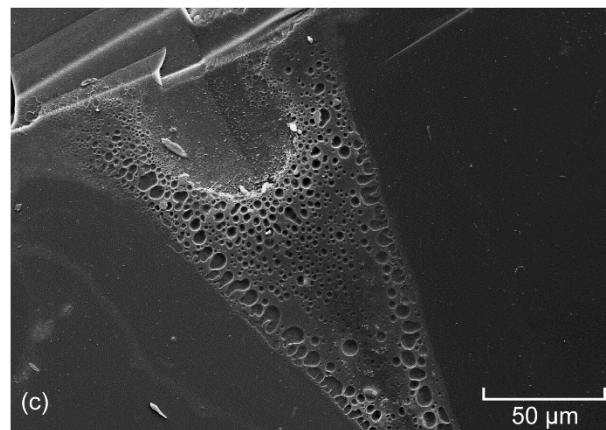
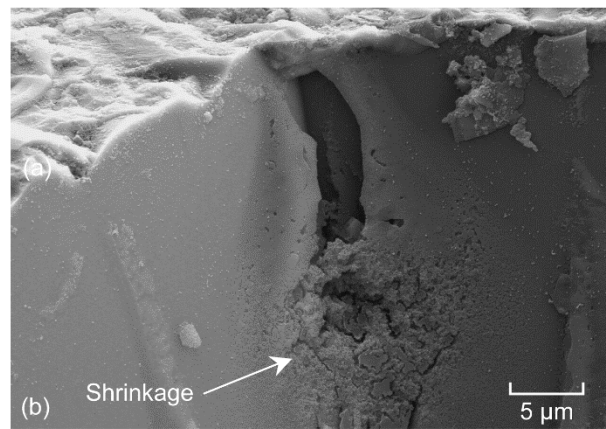
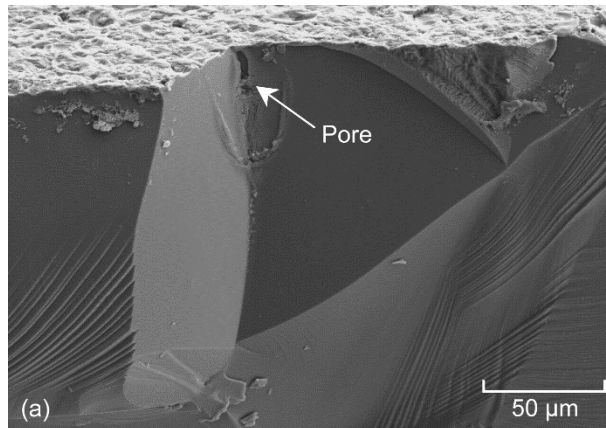


Figure 7.—Porosity observed on fracture surfaces.
 (a) Pore with shrinkage. (b) Closeup of pore with shrinkage. (c) Cluster of pores.

TABLE I.—MEAN FRACTURE TOUGHNESS
AND STANDARD DEVIATION IN MPa√m
WITH THE NUMBER OF TESTS
IN PARENTHESES

Fracture toughness $K_{Ib}(A)$ (MPa√m)	
~40 percent RH	N ₂
2010 batch	
2.12±0.04 (4)	2.18±0.14 (5)
2015 batch	
2.06±0.07 (5)	2.19±0.04 (5)
Both batches	
2.09±0.06 (9)	2.18±0.10 (10)

TABLE II.—MEAN FRACTURE TOUGHNESS
AND STANDARD DEVIATION IN ksi√in.
WITH THE NUMBER OF TESTS
IN PARENTHESES

Fracture toughness $K_{Ib}(A)$ (ksi√in.)	
~40 percent RH	N ₂
2010 batch	
1.93±0.04 (4)	1.98±0.13 (5)
2015 batch	
1.88±0.06 (5)	1.99±0.03 (5)
Both batches	
1.90±0.06 (9)	1.99±0.09 (10)

TABLE III.—SLOW CRACK GROWTH PARAMETERS FOR TESTING IN WATER

Power Law Crack Growth Parameters (water): $v = \frac{da}{dt} = A_1 K_I^{n_1} = A_1^* \left[\frac{K_I}{K_{Ic}} \right]^{n_1}$			
Vintage	n_1	A_1 m/s(MPa√m) ⁻ⁿ	A_1^* (m/s)
2015	36.5±5.2	1.96×10 ⁻¹¹	48.6
2010	32.9±4.9	1.65×10 ⁻¹¹	2.17
Exponential Law Crack Growth Parameters (water): $v = \frac{da}{dt} = A_2 \exp(n_2 K_I) = A_2 \exp\left(n_2^* \frac{K_I}{K_{Ic}}\right)$			
Vintage	n_2^*	n_2 (MPa√m) ⁻¹	A_2 (m/s)
2015	53.2	24.4	3.05×10 ⁻²¹
2010	61.1	28.0	1.05×10 ⁻²³

TABLE IV.—SLOW CRACK GROWTH PARAMETERS IN NASGRO® US UNITS FOR TESTING IN WATER

Power Law Crack Growth Parameters (water): $v = \frac{da}{dt} = A_1 K_I^{n_1} = A_1^* \left[\frac{K_I}{K_{Ic}} \right]^{n_1}$			
Vintage	n_1	A_1 in./hr(ksi√in.) ⁻ⁿ	A_1^* (in./hr)
2015	36.5±5.2	8.66×10 ⁻⁵	6.90×10 ⁶
2010	32.9±4.9	5.17×10 ⁻⁵	3.07×10 ⁵
Exponential Law Crack Growth Parameters (water): $v = \frac{da}{dt} = A_2 \exp(n_2 K_I) = A_2 \exp\left(n_2^* \frac{K_I}{K_{Ic}}\right)$			
Vintage	n_2^*	n_2 (ksi√in.) ⁻¹	A_2 (in./hr)
2015	53.2	26.8	4.32×10 ⁻¹⁶
2010	61.1	30.8	1.49×10 ⁻¹⁸

TABLE V.—SLOW CRACK GROWTH PARAMETERS IN NASGRO® SI UNITS FOR TESTING IN WATER.

Power Law Crack Growth Parameters (water): $v = \frac{da}{dt} = A_1 K_I^{n_1} = A_1^* \left[\frac{K_I}{K_{Ic}} \right]^{n_1}$			
Vintage	n_1	A_1 mm/hr(MPa√mm) ⁻ⁿ	A_1^* (mm/hr)
2015	36.5±5.2	1.24×10 ⁻⁵⁹	1.75×10 ⁸
2010	32.9±4.9	3.07×10 ⁻⁵⁴	7.80×10 ⁶
Exponential Law Crack Growth Parameters (water): $v = \frac{da}{dt} = A_2 \exp(n_2 K_I) = A_2 \exp\left(n_2^* \frac{K_I}{K_{Ic}}\right)$			
Vintage	n_2^*	n_2 (MPa√mm) ⁻¹	A_2 (mm/hr)
2015	53.2	0.770	1.10×10 ⁻¹⁴
2010	61.1	0.887	3.78×10 ⁻¹⁷

TABLE VI.—BRANCH CONSTANTS AS A FUNCTION OF STRESS RATE AND ENVIRONMENT

Rate (MPa/s)	A_B (MPa√m)	Environment
118	7.5±0.6	Dry N ₂
118	7.9±0.9	Water
24	8.0±1.6	Water
0.024	8.5±2.4	Water
0.0024	8.6±0.9	Water
Combined data	8.1±1.3	-----

